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(54) **Process for the purification of oils.**

(57) A process for the purification of oils such as perfluoropolyether oils, silicone oils or oils based on hydrocarbons, which are contaminated with different impurities, in particular, with dispersed solid matter which can be filtered only with difficulty with conventional filters, which process comprises filtration carried out with a filter of the tangential-flow type, the pores of the filter element having a diameter of less than 0.4μm.

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PROCESS FOR THE PURIFICATION OF OILS

The present invention relates to a process for the purification of oils contaminated with different impurities and, in particular, with dispersed solid matter which can be filtered only with difficulty with conventional filters. The process is useful, for instance, for regenerating oils used as fluids in vacuum pumps and in apparatuses of various kinds wherein, during use, the oil is progressively polluted by foreign substances that may be present in the oil either in solution or in the form of finely-dispersed solid matter. In many cases the dispersed solid substances are either in the form of gels or gums or mucilages which may form a film of practically impermeable matter that drastically reduces the filter efficiency of the filters of the conventional type.

It is known from GB-A-1 561 696 that it is very difficult or almost impossible to eliminate fine particles of foreign materials dispersed in non-conductive liquids, such as, for example, oils, by using a film. The British patent discloses an apparatus for the electrostatic purification of oils by using various collecting members for the impurities, inserted between anode and cathode plates, parallel to the electric field created in the apparatus. The value of the applied voltage is determined by the size of the particles and increases as the particles become smaller. This method is, however, not pratical when a large amount of oil has to be purified and, furthermore, it is also very expensive.

Thus, it is an object of the present invention to provide a very simple and efficient process for the purification of oils, which does not require complicated and expensive apparatuses.

The process and apparatus according to the present invention are particularly suitable for the treatment of perfluoropolyether oils which are, for instance, used in vacuum pumps, for thermal tests of electronic apparatuses and in plasma-etching processes employing gaseous agents such as $SiCl_4$, CF_4, Cl_2 or O_2.

Perfluoropolyethers employed for these purposes are, for instance, Fomblin® (registered trademark of Montedison), Krytox® (Du Pont) and similar products known on the market by the name Tyreno® (Klüber) and Barierta®, as well as the perfluoropolyethers produced according to EP-A-148482.

Several methods are known for the purification of perfluoropolyethers contaminated with liquid impurities such as hydrocarbons (mineral oils). Such methods are, usually, based on extraction by means of solvents or on chemical treatment for removing traces of water (US-A-4 178 465). These methods, however, do not solve the problem concerning the removal of solid impurities, in particular, those in the form of a gel.

The process according to the invention is also suitable for the treatment of other kinds of lubricating oils of different chemical nature such as silicone oils and those based on hydrocarbons.

The basic operations involved in the process according to the invention are:

- An optional degassing for removing volatile products, which is preferably carried out by blowing an inert gas such as nitrogen into the oil which is kept at room temperature or at a temperature above room temperature.

- Filtration for removing the solids in suspension, which is carried out by using a filter of the tangential-flow type, wherein the liquid to be filtered is passed onto the filter surface at high speed, thus avoiding the formation of a deposit of solid material on the surface itself. The diameter of the pores of the filter surface has to be very small so as to achieve a complete separation of the smallest solid particles as well. Diameters of less than 0.4 μm and, preferably, in the order of about 0,2 μm, are required.

A suitable type of filter for carrying out the filtration is that produced by Membrana Inc., U.S.A., and marketed under the names DYNA-SEP® and PHARMA-SEP®. The filtering surface of such a filter consists of a cylindrical or tubular element made of porous polypropylene, through which the liquid to be filtered is passed at high speed and is kept at a pressure above that outside the tubular surface. On leaving the tubular filtering surface, the filtrate is collected while the liquid to be filtered is recycled continuously until the concentration of dispersed solid substances reaches a high value. The pressure of the liquid to be filtered obviously depends on the characteristics of the apparatus which may, at the most, be operated at a pressure of up to 4 bar.

The temperature of the liquid is obviously an important factor as well. A certain temperature limit cannot be exceeded. This is determined by the thermal resistance of the material of the filter element. For the filters described above, this temperature limit is 60°C.

The process according to the invention may also comprise accessory, additional operations such as a previous decantation for a preliminary separation of coarser solids and of liquid, immiscible impurities, if present, and a pre-

ceding filtration on a filter capable of separating the coarser particles in order to lower the solids content of the liquid to be conveyed to the tangential-flow filter.

After the filtration in the filter of the tangential-flow type according to the present invention, the perfluoropolyether or the mineral oil can be used directly as a fluid in vacuum pumps or it can be subjected to neutralization in order to eliminate acid or basic substances that might be present. In particular, the neutralization of oils having acid impurities can be carried out with a cartridge charged with activated alumina, activated carbon or sodium carbonate.

The following examples illustrate the purification of a few types of oil in accordance with the present invention.

EXAMPLE 1

Product to be purified: 12 kg Fomblin® Y L VAC 25/5 oil used in pumping assemblies of an apparatus for plasma-etching with $SiCl_4$, CF_4, Cl_2, O_2 as gaseous agents. The product, having a light-brown colour, contained solid substances dispersed in the form of a gel. After decantation over a period of three weeks no separation of distinct phases was achieved. The product, kept at 50°C, was degassed in a heated metal container by blowing 135 l/h of nitrogen into the product for 4 hours.

The degassed product was then subjected to a pre-filtration with a filter having a pore diameter of 15 µm, at a pressure of 1.7 bar.

Filtration with a DYNA-PHARMA® filter which had a filter element with a pore diameter of 0.2 µm, a filter cartridge

of about 51 cm in length and a filtering surface of about 19.5 dm^2 was started.

Operating conditions: Filtration temperature 50 to 55°C; inlet pressure 2.2 bar; outlet pressure 0.8 to 0.9 bar.

The filtration was carried out until 11.3 kg of oil had been recovered which corresponded to 94% by weight of the starting product. The average filtration rate was 11.5 1/h.

EXAMPLE 2

Product to be purified: about 20 kg of Fomblin® Y L VAC oil coming from an apparatus for ion implantation and contaminated, inter alia, with PH_3 and AsH_3.

Decantation: After two weeks a distinct liquid phase having a brown colour was formed. The main phase was separated and pre-filtration with a filter consisting of cellulose nitrate having a pore diameter of 25 µm was started. The liquid to be filtered was kept at 60°C.

The pre-filtered liquid was then conveyed to a tangential filter having a filter cartridge as described in example 1. The filtration temperature was 60°C, the inlet pressure 2.2 bar and the outlet pressure 0.8 to 0.9 bar.

An average filtration rate of 7.7 1/h was noted.

COMPARATIVE EXAMPLE

The pre-filtered liquid was subjected to filtration on a vacuum filter of the conventional type with a filtering membrane made of cellulose nitrate having a pore diameter of 0.8 µm. The filtration rate decreased very quickly and, after 1 hour, the filter was completely clogged and

the amount of collected filtrate was negligible.

EXAMPLE 3

The product to be purified consisted of 5 kg of Wacker AN 30® silicone oil used in pumping assemblies of an apparatus for plasma-etching, utilizing CCl_4, $SiCl_4$, O_2, N_2 as gaseous agents. The product had a light-brown colour and, although it contained dispersed solid matter in the form of a gel, no separation of distinct phases had been achieved after three weeks.

The product, kept at 50°C, was degassed in a heated metal container by blowing 135 l/h of nitrogen into the product for 4 hours.

Then filtration with a PHARMA-SEP® filter of about 25 cm length and a filtering surface of 6.32 dm^2, having a filter element with a pore diameter of 0.2 µm was started. Operating conditions: Filtration temperature 50°C, inlet pressure 1.5 bar, outlet pressure 1 bar, average filtration rate 3 l/h.

The filtration was repeated with the PHARMA-SEP® filter described above. However, the working conditions were changed as follows: inlet pressure 1.8 bar, outlet pressure 1.2 bar, filtration temperature 50°C. The average filtration rate was found to be about 3.6 l/h.

In both test series the filter did not give any indication of obstruction.

EXAMPLE 4

The product to be purified consisted of 5 kg of Edwards 15 mineral oil used in pumping assemblies of an apparatus

for ion implantation utilizing PH_3, AsH_3 and N_2 as gaseous agents. The product had a dark colour and contained solid matter dispersed in the form of darkly-coloured impurities and of gels.

The product, kept at 50°C, was degassed in a heated metal container by blowing 135 l/h of nitrogen into the product for 4 hours.

Then filtration with a PHARMA-SEP® filter of the same type as that used in example 3 was started. Operating conditions: filtration temperature 50°C, inlet pressure 1.6 bar, outlet pressure 1 bar, average filtration rate 1.2 l/h.

Filtration was repeated with the same PHARMA-SEP® filter described above, however, the working conditions were changed as follows: inlet pressure 1.4 bar, outlet pressure 0.8 bar, filtration temperature 50°C. The average filtration rate was found to be about 1.1 l/h. In both test series the filter did not give any indication of obstruction.

CLAIMS:

1. A process for the purification of oils such as perfluoropolyether oils, silicone oils or oils based on hydrocarbons which comprises removing by filtration impurities, in particular, dispersed solid matter which can be filtered only with difficulty by conventional filters, characterized in that said filtration is carried out by means of a filter of the tangential-flow type, wherein the pores of the filter element have a diameter of less than 0.4 μm.

2. A process according to claim 1, wherein a degassing treatment by introducing a flow of inert gas and/or a pre-filtration on a conventional filter is carried out before filtration on the tangential-flow filter.

3. A process according to claim 1 or 2, wherein the tangential-flow filter is provided with a filter element having a pore diameter of about 0.2 μm.

4. A process according to any one of claims 1 to 3, wherein the filter element consists of porous polypropylene.

5. A process according to any one of claims 1 to 4, wherein the oil to be purified is subjected to previous decantation so as to remove liquid impurities immiscible with the oil, if present.